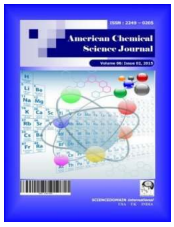

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Synthesis and Characterization of Crude and Oil Extract of Paracress (*Spillanthesacmella*)

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Authors' contributions

This work was carried out in collaboration between all authors. Author MFA designed the study, performed the analysis and wrote the protocol. Author OAO supervised the work. Authors SA and ATO managed the analyses of the study and the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Syhthesis and characterization of crude and oil extract of *Spillanthesacmella* was carried out using petroleum ether, chloroform and methanol. The extraction of *Spilanthesacmella* oil and crude extract from powdered *Spilanthesacmella* plant using petroleum ether gave 1.2406 g, chloroform gave 0.6412 g and methanol gave 8.4231 g. 5.049 g of *Spilanthesacmella* oil was gotten and 2.4341 g of potassium chloride was collected from the oil. The result showed the presence of amine, alkene, ketone/aldehyde, carbonyl and ester at different frequencies which is in line with other works done. Deca-6, 8 dinyoic acid isobutylamide was isolated using IR, 1HNMR and 13C-NMR spectra.

Keywords: Spilanthesacmella; characterization; synthesis.

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1. INTRODUCTION

Spillanthesacmella is a heavy feeder plant, preferring rich soils and an occasional side dressing of organic compost seed, whole plant and stem may be propagated. About (84) Eight-four species of Spilanthes have been reported from various part of the world. The genus occurs widely in damp pastures, at swamp margins, on rocks near the sea and as a weed on road sides. The *Spilanthesacmella* plant have a pungent taste when chewed, a strong, spicy, warmth spread outward across one's tongue, turning into a prickling sensation. With this, the salivary glands leap into action, pumping out quantities of saliva. As the prickling spreads, it mellows into an acidic sharpness accompanied by tingling and then numbness. The numbness fades away after some time (2 to 20 minutes) depending on the person and amount eaten and the pungent after taste may linger for an hour or more [1,2,3].

Spilanthesacmella has a long history of use as a folklore remedy e.g. for toothaches, rheumatism, fever, etc. [4]. The plant has found applications in pharmaceuticals, as an anti-toothache formulation for pain relief, swelling and gum infections, periodontosis and mouthwashes [5,6]. In addition, its extracts is an active component added to body and beauty care cosmetics as a fast acting muscle relaxant to accelerate repair of functional wrinkles [7], the plant extract was also used for stimulating, reorganizing and strengthening the collagen network in anti-age-applications for example in antiwrinkle cream formulation [8,9].

As a nutritional supplement [10], small amounts of a plant extract have been used for taste improvement as a sweetener with high sweetness devoid of unpleasant smell after the taste or odour of foods or drinks [11].

A number of constituent had been isolated from the *Spilanthesacmella* for example spilanthol, isobutylamides, Vanilic acid, [12,13,3,]. *α*-and *β*-amyrin esters, stigmasterol, myricyl alcohol including sitosterolglucosides [14] and triterpenoidal saponins [13]. Spilanthol showed interesting bioactivities, e.g. strong local anesthetic [15], analgesic [16,17] and insecticidal activities [4]. Crude flower head extracts of the plant exhibited potent ovicidal, marked larvicidal (LC_{50} of 61.43 ppm) and pupicidal activities [18,19]. The extracts were also shown to exhibit pancreatic lipase inhibition which has potential as candidates for weight loss and obesity control [20]. The study was carried out for synthesis and characterization of crude and oil extract of the plant.

2. MATERIALS AND METHODS

2.1 Plant Collection and Identification

Spillanthesacmella (fresh leaves) was collected from Ikare Akoko in Ondo state. The plant was identified and authenticated by a botanist and was thoroughly washed with water to remove sand and other debris before being dried on the bench at room temperature to avoid the destruction of the active ingredient of the plant. The dried plant was then mashed using mortar and pestle, then stored in dry airtight containers before extraction proceeds. Some of the solvents used were purified by distillation before used while the solids reagents were used directly without any further purification. All the equipment were thoroughly washed with soap and water and then dried in the over before they were used.

2.2 Preparation of the Aqueous Extract

46 g of powdered *Spilathesacmella* sample was placed in a porous thimble chamber, extracting solvent in the boiling flash. The solvent was heated on water bath to reflux and the distillate as it drops from the condenser collected in the thimble chamber. As the solvent came in contact with the solid in the thimble, the liquid (i.e. extracting solvent) effect the extraction. After the chamber had been filled to upper level of siphon aim, the solution was emptied into the boiling flash by siphoning action. The process was allowed to siphon for about 5 – 6 times. The extracted mixture was collected in the boiling flash. The solution was subjected to continuous distillation under reduced pressure and the condensed distillate returned as fresh extracting solvent to the extraction vessel and reused [1]. The extract was concentrated and put in a specimen bottle at room temperature until used.

2.3 Distillation

This is a process by which a distillation was carried out below it normally boiling temperature to prevent denature of the extract. 1000 ml round bottomed flask containing the sample extracted mixture connected to a condenser was set up on a water bath, the condenser at the other end was connected to another round bottomed flask, immersed in an ice connected to a vacuum pump

and the solvent was distilled off. The residue containing *Spilanthesacmella* extract was poured out into a 100 ml beaker to allow the residual solvent to evaporate. The same methods were repeated for all the solvent used for the extraction. For the petroleum ether and chloroform, extraction and distillation was carried out using a very cold ice water for condensation to prevent loss of solvent due to their low boiling point.

2.4 Determination of Elemental Constituents

Various fractions of the methanol extracts were eluted using different elution with different solvent system prepared from solvent mixtures from petroleum ether, chloroform, ethyl acetate and methanol. The eluents were concentrated and each fraction (component) was subjected to thin layer chromatography.

The spectra properties of *Spilanthesacmella* extract and its modified derivatives were done using Infra-Red Spectrophotometer Model B UCK 500 M and Nuclear Magnetic Resonance Spectrophotometer of Model 200 BB. The result of the experiment is as presented in Tables 1-3.

Table 1. Some isolated compounds from methanol extracts and oil

Extract	Solvent used	Mass of compound
A	Petroleum ether(60-80)	0.8124 g
B	5:1 ethyl acetate-methanol	1.9012 g
C	5:1 ethyl acetate-methanol	1.0052 g
D	5:1 ethyl acetate-methanol	1.2412 g
E	Methanol	5.1240 g

A = fraction 1,2,3, are the same;
B = fraction 19;
C = fraction 26;
D = fraction 27;
E = purified oil

3. DISCUSSION

The extraction of *spilanthesacmella* oil and crude extract from powdered *spilanthesacmella* plant using petroleum ether gave 1.2406 g, chloroform gave 0.6412 g and methanol gave 8.4231 g. 5.049 g of *spilanthesacmella* oil was gotten and 2.4341 g of potassium chloride was collected from the oil.

Some isolated compounds from methanol extract and the oil obtained from column Chromatography are presented in Table 1 while the Rf values of some isolated compounds of methanol extract are as presented in Table 2. The spectra average frequencies and functional group types of each of the partially purified isolated compounds from methanol extract and purified oil of methanol extracts are presented in Table 3, (A to D) gave the IR spectra of isolated compounds from methanol extract and E gave the IR spectra of isolated compound from methanol extract oil.

Table 2. Rf values of some isolated compounds of methanol extract

Isolated compounds	TLC solvent	RF values
A	3:1 petroleum ether-ethyl acetate	0.79
B	Ethyl acetate	0.72
C	Ethyl acetate	0.59
D	Ethyl acetate	0.64
E	Methanol	0.45

Isolated compound from the methanol extracts are presented in Table 1. Sample A indicate the following functional group at 3442.45 cm-1, (N-H in amine), 2926.85 cm-1 (C=C-H in alkenes) and 1714.77 cm-1 (C=O in aldehyde or ketones). Sample B suggest the following functional groups at 3424.04 cm-1, (N-H in amine), 2964.68 cm-1, (C=C-H in alkenes), 2932.99 cm-1, (C=C-H in alkenes), 2362.14 cm-1, (-C=C- in alkenes), 1717.84 cm-1 (C=O aldehyde or ketones) and 1656 cm-1 are (C=O in carbonyl). Sample C indicate the following functional groups 3417.90 cm-1, (N-H in amine). 2964.68 cm-1, (C=C-H in alkenes) 1714.77 cm-1 (C=O aldehyde or ketones), 1371.28 cm-1, (C-O in ester).

Sample D present the following functional groups at 3417.90 cm1 (N-H in amine). 2964.68 cm-1, (C=C-H in alkenes), 1714.64 cm-1 (C=O aldehyde or ketones), while Sample E, which is the purified oil indicate the following functional group at 3374.93 cm-1, (N-H in amine) 2945.26 cm-1, (C=C-H in alkenes)) 2362.14 cm-1, (-C=C- in alkenes) 2073.65 cm-1, (C=C in alkenes) and 1631.97 cm-1. (N-C=O in amide).

Table 3. Frequencies (Cm^{-1}) and functional groups of isolated compounds

Extract: Isolated compounds	Frequencies (Cm^{-1})	Functional group
Sample A		
(a)	3442.45	N-H
(b)	2926.85	C=C-H
(c)	2364.14	C=C
(d)	1714.77	C=O
Sample B		
(a)	3424.04	N-H
(b)	2964.68	C= C-H
(c)	2932.99	C=C-H
(d)	2362.14	C=C
(e)	1717.84	C=O
(f)	1656.50	C=O
Sample C		
(a)	3417.90	N-H
(b)	2963.68	C= C-H
(c)	1714.77	C=O
(d)	1371.28	C-O
Sample D		
(a)	3411.76	N-H
(b)	2963.68	C-H
(c)	1714.77	C=O
Sample E		
(a)	3374.93	N-H
(b)	2945.26	C=C-H
(c)	2362.14	C=C
(d)	1631.93	N-C=O (amide)

4. CONCLUSION

An alkamide, deca-6, 8-diynoic acid isobutylamide was isolated from *Spilanthesacmella* plant. The structural elucidation of the above compound was achieved from the IR, 1HNMR and 13C-NMR spectra. Experiments and the comparison of the spectral data with published value showed that this structure had not been reported else where. The IR spectra showed the following isolated functional groups. 3374.93 cm-1, (N-H in amine) 2362.14 cm-1 (C≡C in alkynes) and 1631.97 cm-1 (N-C=O in amide).

The 13CNMR Spectrum of the isolated compound presents the following:

At 160.978 ppm (C=O in carbonyl) was noticed. At 77.771ppm to 74.978 ppm, 2(C≡C in alkynes) was noticed. At 50.691 ppm, 29.635 ppm-26.399 ppm, 5(CH_2 in alkyl) was indicated. At 24.729 ppm to 22.606 ppm 2(CH) was noticed, at 20.776 ppm to 18.741 ppm, 2(CH_3 in alkyl) was indicated.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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